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EXTRUSION OF TUNGSTEN- UO_2 HONEYCOMB STRUCTURES (U)

by

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ABSTRACT

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This project was undertaken to investigate the application of the coextrusion process to the fabrication of tungsten and W-UO₂ in a hexagonal honeycomb geometry. Extrusions of tungsten with molybdenum as the sacrificial material were performed. One extrusion of W-UO₂ was also made. The effects of billet component fabrication history, billet cleaning methods, and extrusion parameters on geometry control and W-W bonding are discussed. Conclusions reached and recommendations for future work on the process are outlined.

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EXTRUSION OF TUNGSTEN- UO_2 HONEYCOMB STRUCTURES

INTRODUCTION

The requirement for high temperature and high heat flux in nuclear rocket reactor fuels has dictated the development of refractory metal base fuel elements with a high surface to volume ratio. In the water moderated nuclear rocket concept being developed by the NASA-Lewis Research Center, fuel material comprising UO_2 uniformly dispersed in a tungsten matrix will be used in the fuel elements. Sufficient testing of this fuel material has been completed to establish it as a promising candidate for applications at operating temperatures to 2500 C in a hydrogen atmosphere. In the course of this testing it has been determined that a thin tungsten cladding is required to prevent loss of UO_2 from the fuel element at high temperature. A major problem to be solved is that of fabrication of tungsten clad W- UO_2 into a geometry which is structurally stable in a high temperature-high velocity hydrogen atmosphere. A honeycomb structure appears to adequately meet all the criteria.

Present state of the art methods of honeycomb fabrication do not easily lend themselves to the manufacture of tungsten clad W- UO_2 fuel elements due to the problems of forming and joining the brittle material. A process utilizing coextrusion was developed by Pacific Northwest Laboratories for the fabrication of honeycomb shapes of Zircaloy clad uranium metal.¹ This process offered a possibility of application to the W- UO_2 material. The process as shown in Figure 1 consists essentially of fabricating an evacuated extrusion billet of the desired materials in the desired cross sectional geometry with a sacrificial material filling those areas which are vacant in the final product. This billet is hot extruded through a round die to reduce the cross section to the final area and to simultaneously bond all components. The extrusion is cut to length and

¹ G. S. Allison. The Field of High Surface Area to Fuel Volume Ratio Nuclear Fuel Elements of a Wide Range of Materials and Operating Temperatures, Invention Report HWIR-1645, AEC Patent Case No. S-29356, U. S. Patent Application No. 374,855, filed June 12, 1964.

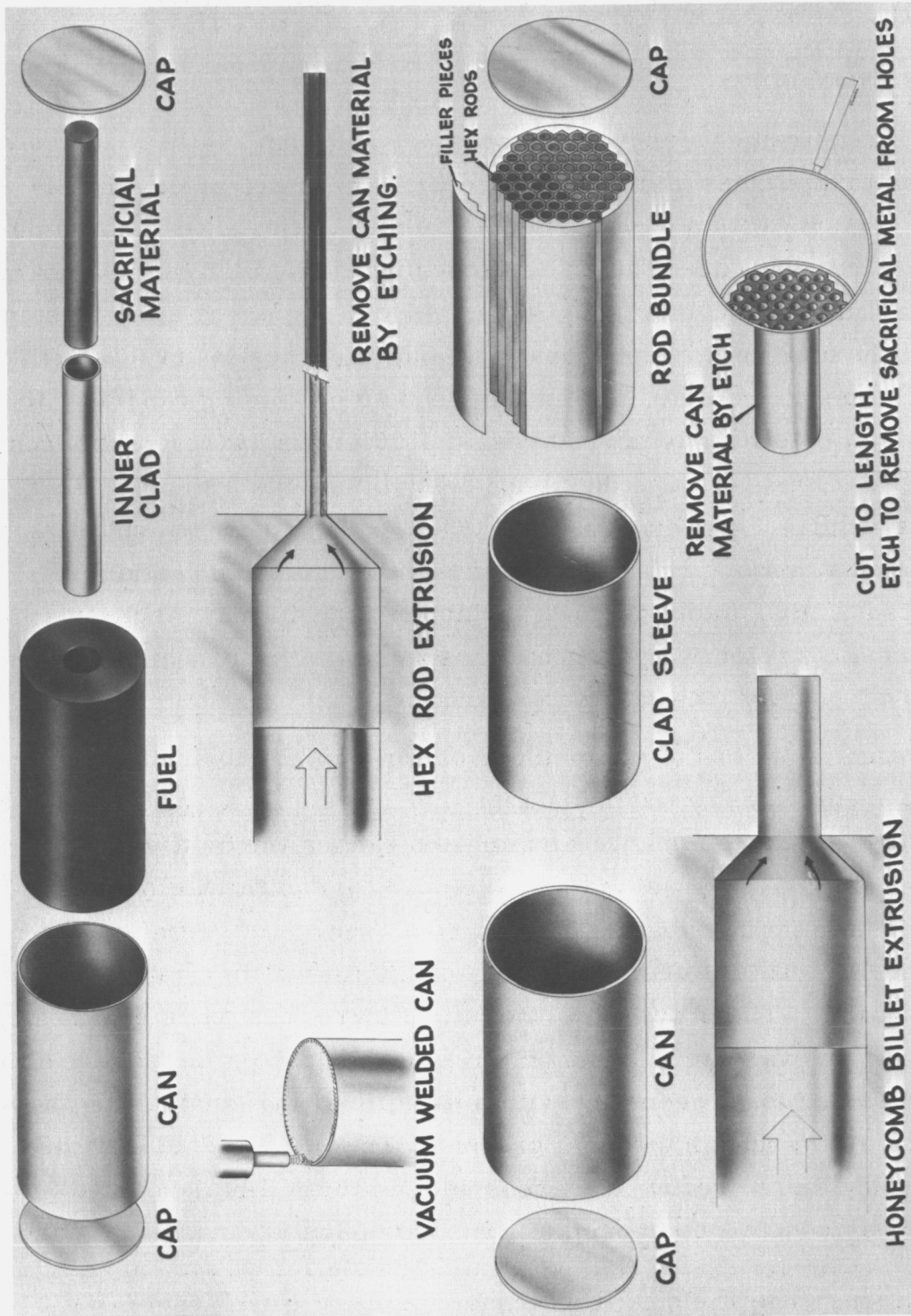


FIGURE 1

Honeycomb Fuel Fabrication Process

the sacrificial material chemically removed to leave the clad honeycomb fuel shown in Figure 2. The cut ends are subsequently closed, if necessary, by welding, brazing, or vapor coating. Control of the shape of the honeycomb cells is subject to the variations in flow inherent in the extrusion process. The two dimensions considered most critical, cross sectional areas of coolant passages and fueled webs, can be very closely controlled. The external dimensions and shape of the fuel element can also be controlled within acceptable tolerances. The end product is wrought, fully bonded honeycomb with a clad, fueled web of uniform thickness, and coolant flow passages of uniform area.

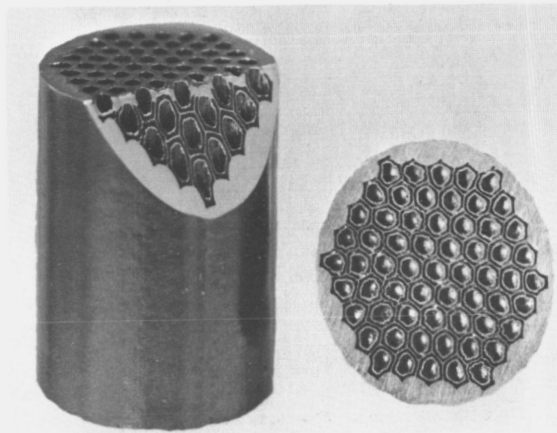


FIGURE 2

Zircaloy-2 Clad Uranium
Honeycomb Fuel

(1.0 in. diameter Zircaloy-2 clad uranium metal honeycomb fuel with braze closed ends. The section on the right shows the extrusion prior to closing and sacrificial material removal.)

The good results attained from this process in fabricating Zircaloy-clad uranium honeycomb was the basis for investigating the feasibility of its application to tungsten-clad tungsten- UO_2 honeycomb. This program was sponsored by the National Aeronautics and Space Administration. The first phase of the program was to attempt to fabricate unfueled tungsten honeycomb with approximately 1.5 in. outside diameter, 0.020 in. thickness, and coolant flow passages of 0.125 in. hydraulic diameter. The cross section of this honeycomb is shown in Figure 3. Successful completion of this phase would lead to the fabrication of similar geometries with a UO_2 fueled web.

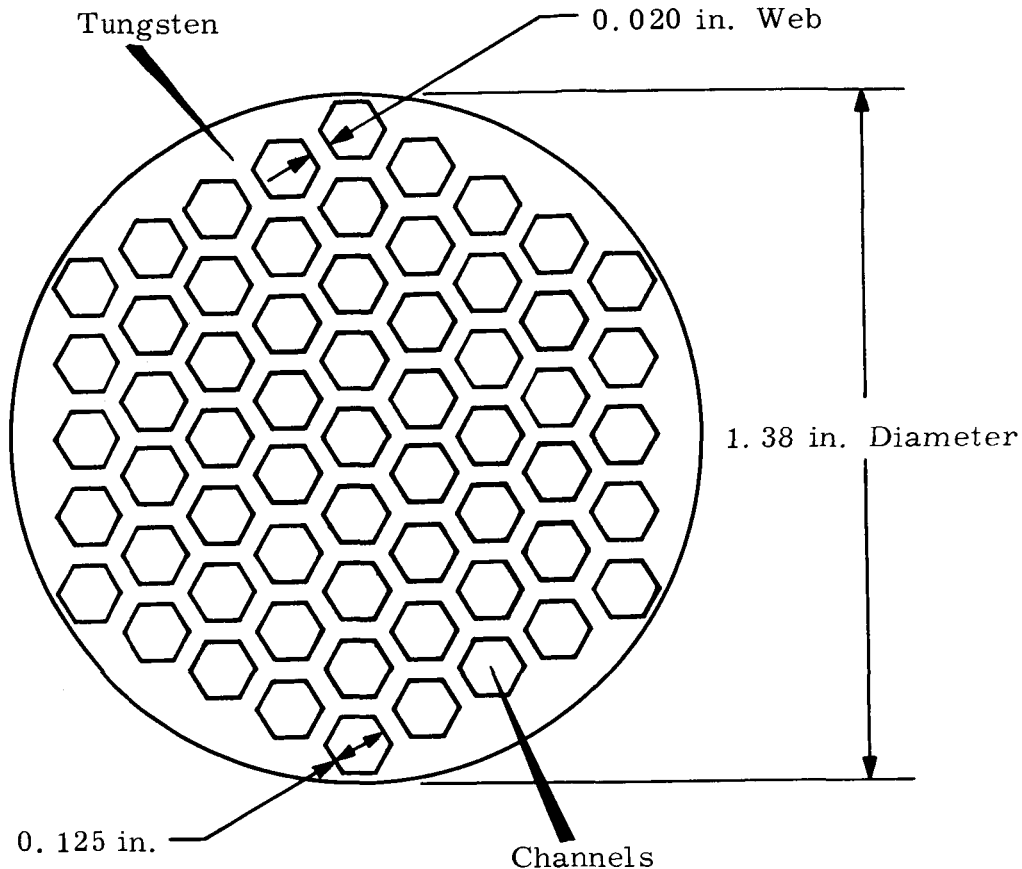


FIGURE 3

Shape and Approximate Dimensions of Desired Unfueled
Honeycomb Cross Section

The initial stage of the development was devoted to determining the effective characteristics of tungsten and molybdenum when applied in the honeycomb extrusion process. The first experiments were performed to determine the billet component preparation and extrusion parameters required to produce an adequate tungsten-tungsten bond. The effects of the billet component fabrication history and the extrusion parameters on the grain structure and geometry uniformity were concurrently observed in these experiments. After arriving at a satisfactory set of conditions to produce tungsten-tungsten bonding the following approach was selected to fabricate the tungsten honeycomb:

1. Coextrude tungsten-clad, hexagonal molybdenum rod.
2. Machine a molybdenum billet can and tungsten clad the inside diameter by vapor deposition to form the outer shell of the honeycomb.
3. Assemble the rods into the can with the tungsten filler pieces necessary to make the transition from a hexagonal honeycomb array to the circular outer shell, and vacuum close.
4. Extrude this composite billet to bond all tungsten-tungsten interfaces and reduce to the desired final geometry.
5. Cut to lengths and chemically remove the molybdenum to leave the tungsten honeycomb.

The latter period of the program was devoted to investigating possible methods of improving the uniformity of tungsten honeycomb geometry.

Extrusion of W- UO_2 (with an initial UO_2 particle size of 30 - 60 μ) by the Lewis Research Center¹ indicated that excessive stringering of the UO_2 would be a deterrent to the success of this process. A possible solution to this problem is the use of coprecipitated tungsten-submicron UO_2 powder.² One extrusion of this powder was performed with encouraging results and is discussed later in this report. A summary of the extrusions performed in the program is presented in Table I and described in detail in this report. Program support was terminated by NASA prior to completion because other fabrication methods later showed promise of success without the problem of UO_2 stringering.

¹ C. P. Blankenship and C. A. Gyorgak. Preliminary Investigation of Extruding Composite Tungsten- UO_2 Tubing, NASA-TMX-1072, Lewis Research Center.

² R. V. Bowersock. Submicron Dispersion of UO_2 in Tungsten, NASA-CR-54138 (to be published).

TABLE I
EXTRUSION DATA SUMMARY

Number	Billet Type	Material	Billet Diameter, in.	Billet Lubricant	Preheat, °C	Maximum Extrusion Constant, * tsi
MoW-1	1	Mo-W	2.25	Pyro-graphite	2200	19.5
MoW-2	1	Mo-W	2.25	Pyro-graphite	1850	24.0
MoW-3	1	Mo-W	2.25	Plain graphite	1800	24.0
MoW-4	2	Mo-W	2.25	None**	1850	35.5
MoW-5	3	Mo-W	3.00	None**	1800	30.0
Powder 1	4	Wpowd-SS	2.25	Plain graphite	1200	25.0
Powder 2	4	Wpowd-MS	2.25	Plain graphite	1200	23.0
Powder 3	4	Wpowd-MS	2.25	Plain graphite	1100	24.0
Powder 4	5	W-UO ₂ powd	2.25	Plain graphite	1875	36.8
Powder 5	6	W-Mo powd	2.25	Plain graphite	1800	36.8***
Coated Cont. 1	7	304 SS	3.00	Plain graphite	1200	23.9
Coated Cont. 2	7	304 SS	3.00	Plain graphite	1150	26.3

Note: ZrO₂ coated dies used on all but MoW-1.

Ram speed 60 in. /min

Reduction ratio approximately 9 to 1

SS = Stainless Steel

MS = Mild Steel

* Extrusion constant = pressure/ln (reduction ratio)

** Oil-graphite dispersion coating in container

*** Stalled after 7 in. of extrusion

Billet Type

1. Mo can-W sleeve-W sleeve-Mo core
2. Mo can-W sleeve-W plates-Mo spacers
3. Mo can-W coated, hex, Mo rods
4. SS or MS can-W powder core
5. Mo can-W-UO₂ powder core
6. Mo can-co-compacted hex Mo powder rock in W powder matrix
7. Solid billet

EXPERIMENTAL PROCEDURE

A 700 ton, vertical, self-contained hydraulic press equipped for extrusion (Figure 4) was used in this program. It has speeds of 0 to 60 in. /min for pressing and 800 in. /min for approach and return. Stroke and daylight are 36 and 60 in. , respectively. The container is manipulated by a 75 ton, 18 in. stroke hydraulic cushion cylinder mounted in the bed. The pneumatic elevator, which transfers the extrusion from the pit to the floor, was modified to permit extrusion into a vermiculite-filled tube to prevent rapid quenching. Only minor tooling modifications, as later described, were required for this program.



FIGURE 4

Press Used for Extrusion Work in This Project

A vertical coil, argon atmosphere, 2200 °C, induction furnace with a pneumatic, bottom loading elevator was constructed as shown in Figure 5. It was powered by an existing 3000 cps, 200 kW induction generator. All other supporting equipment was available and required no modification.

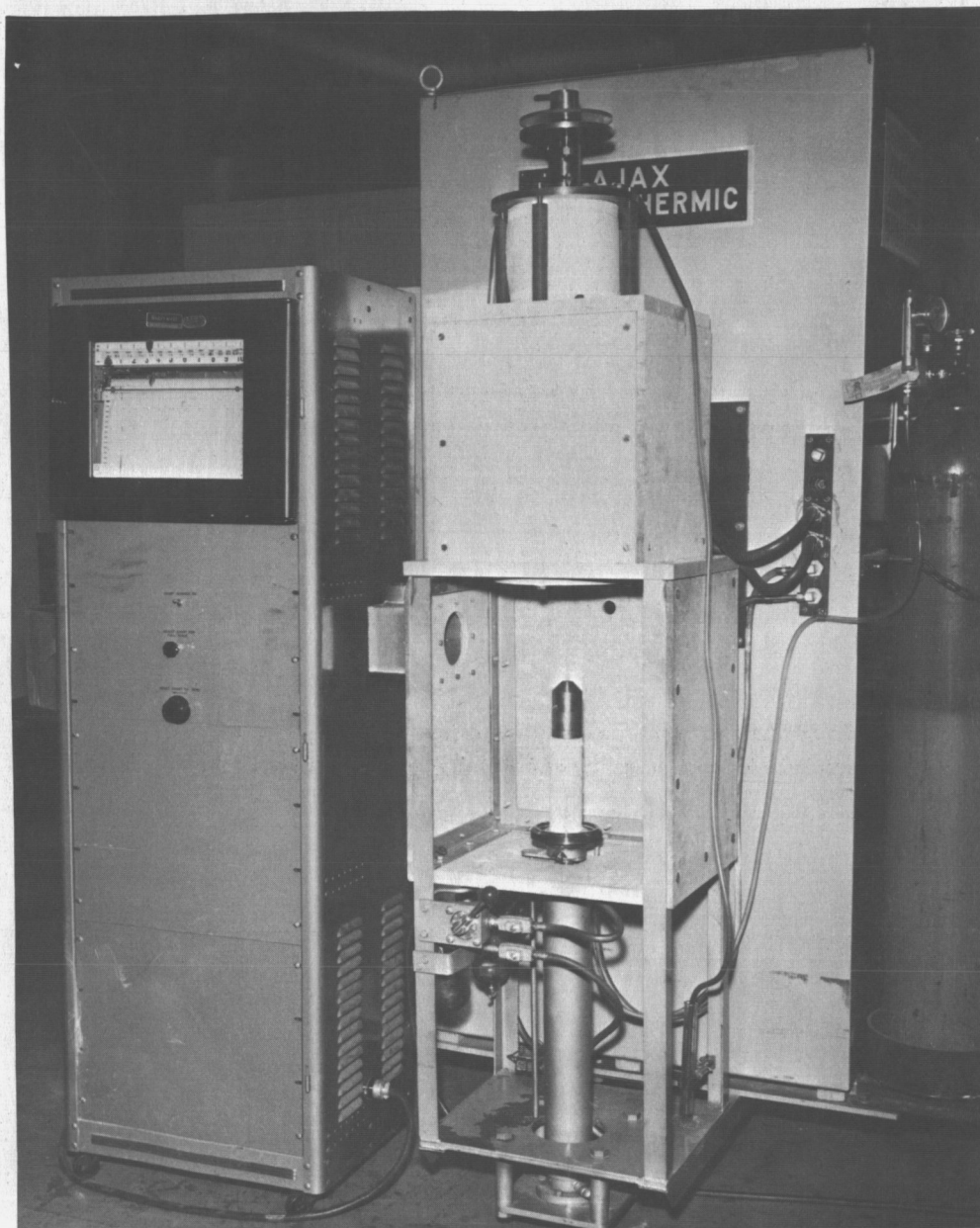


FIGURE 5

3000 Cycle Induction Furnace Used for Preheating Billets
to 2200 °C Maximum in Argon Atmosphere

The first experiments were designed primarily to determine the billet component cleaning technique, billet preheat temperature, and extrusion ratio which were necessary for adequate W-W bonding. Commercial pressed and sintered molybdenum and tungsten powder components were procured to make up composite billets as shown in Figure 6. The 0.125 in. wall nested tungsten sleeves with a molybdenum can and core were selected as the simplest geometry which would furnish the desired data. A 2.25 in. diameter, 90° entrance cone die (9:1 ratio) was arbitrarily selected for this set of experiments.

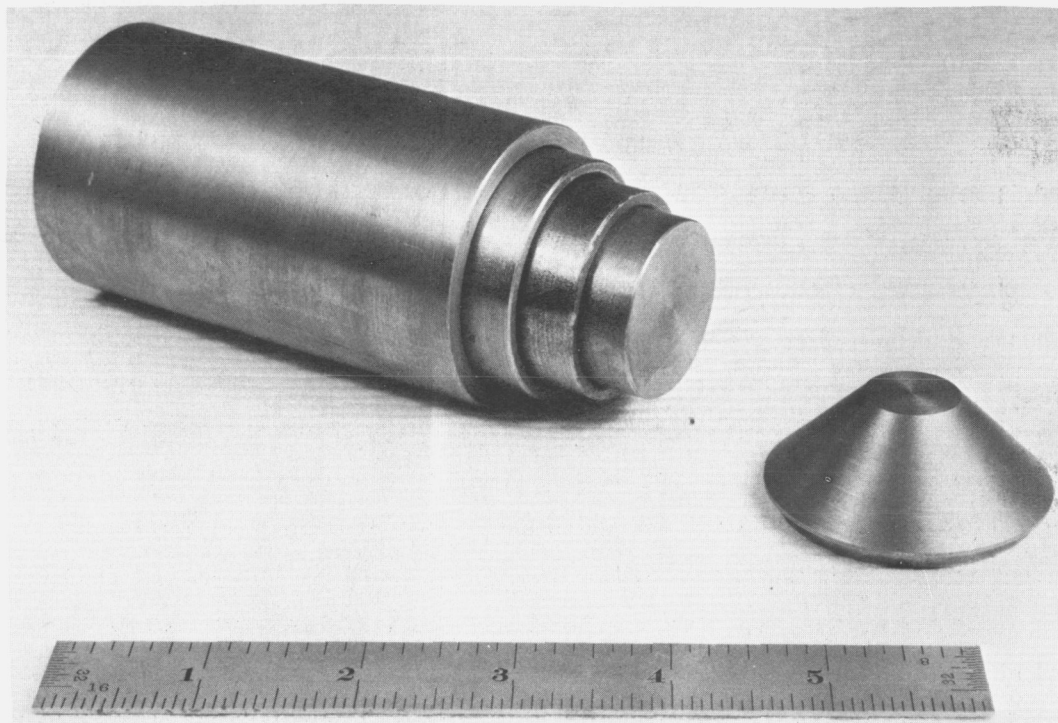


FIGURE 6

Extrusion Billet Used for Extrusions MoW-1, 2, and 3

The 60 in. /min pressing speed available was considered extremely low for refractory metal extrusion and would require high billet preheat temperatures. The high temperature billet would require good thermal protection of the container and die. Pyrolytic graphite, with its low thermal

conductivity and good lubrication properties, was selected for this purpose. Sleeves with an integral cone and a 0.035 in. wall were procured.

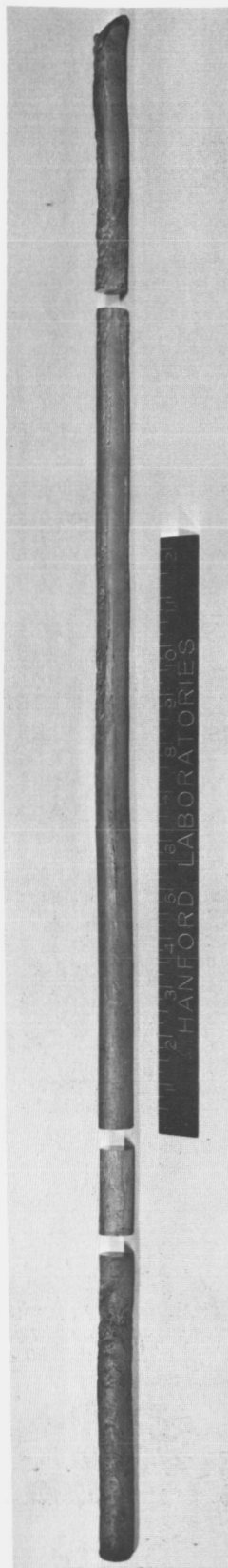
The components for the first extrusion, MoW-1, were cleaned by etching in HNO_3 -5% HF and closed by vacuum electron beam welding. The uncoated T-1 tool steel die with a 0.750 in. diameter throat and a pyrolytic graphite sleeve were placed in position in the 500 °C container to preheat. An uncoated die was used to determine whether the graphite would afford thermal protection for the die. The billet was preheated to 2200 °C in argon in 25 min. plus a 5 min. soak at temperature. Transfer of the billet from the induction furnace to the press, which in all cases is manual, was delayed 10 sec. due to the billet's sticking to the furnace pedestal and having to be broken free. Total time from out-of-furnace to application of load was 20 sec. It was extruded at 60 in./min ram speed requiring 170 tons maximum and 140 tons minimum force. The uncoated die was severely washed to about 1 in. diameter, and the extrusion surface was correspondingly extremely rough as shown in Figure 7(a). Metallography indicated a melting reaction combining iron, carbon, and molybdenum had occurred after the first 6 in. of extrusion, probably due to the breakup of the pyrographite sleeve. The tungsten sleeves showed equiaxed grains varying in size from 0.02 mm at the front to about 0.002 mm at the rear. Approximately 50% of the W-W interface showed complete bonding with grain growth occurring across the interface. The remainder of the interface was cracked and showed evidence of a billet leak or incomplete billet cleaning. The tungsten sleeves were cracked internally in several places. Figures 8 and 9 show the structure and bonding which is better toward the rear of the extrusion.

The second extrusion, MoW-2, billet components were cleaned by cathodic etching in argon and were assembled and vacuum electron beam welded without being reintroduced to the atmosphere. The same general extrusion procedure was followed. The preheat temperature was reduced to 1850 °C and the container temperature was increased to 540 °C. A ZrO_2 coated, H21 tool steel die, 0.730 in. diameter was used. Furnace-to-press transfer time was 10 sec. Maximum force required during the



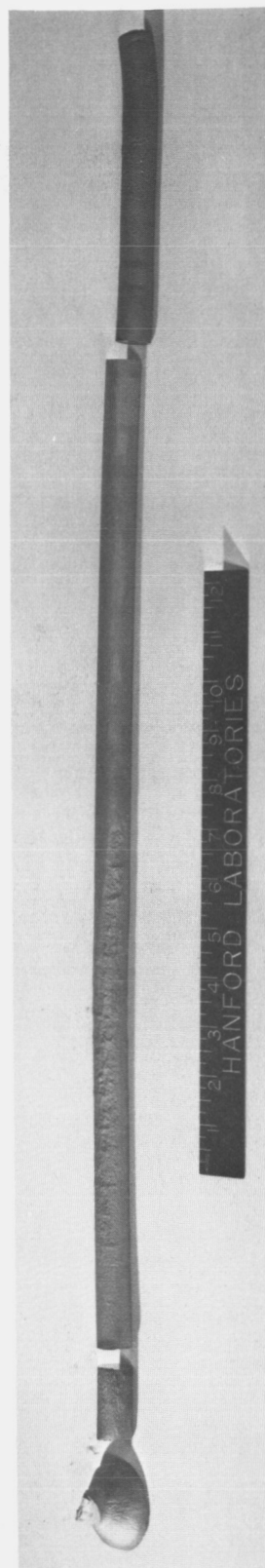
(a) MoW-1

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(b) MoW-2

Neg. 0632612-1

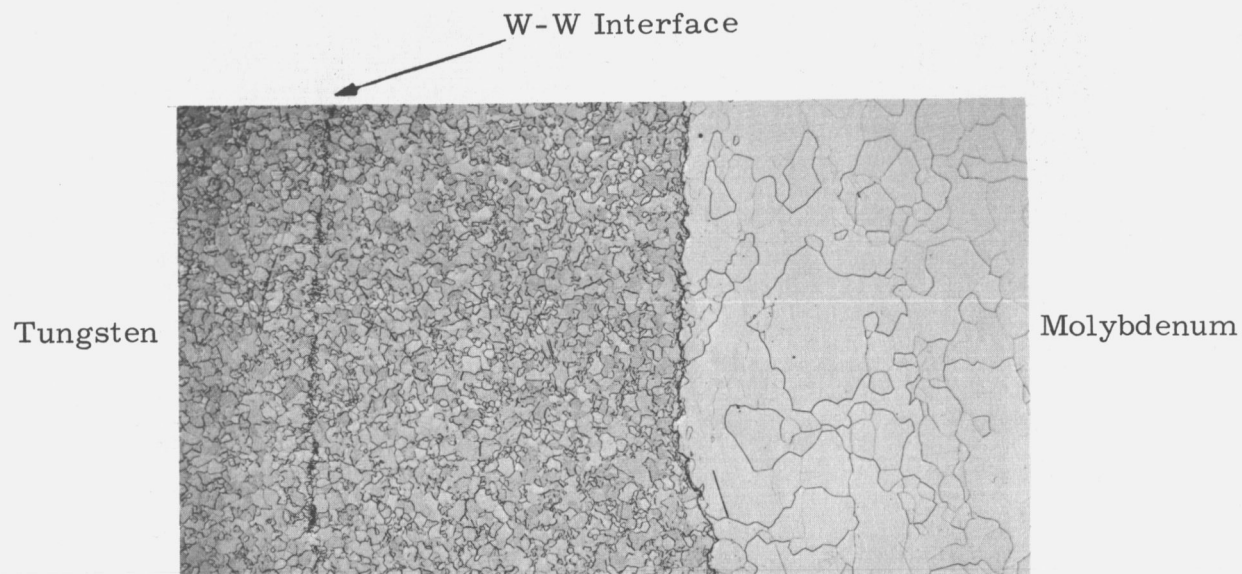


(c) MoW-3

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FIGURE 7

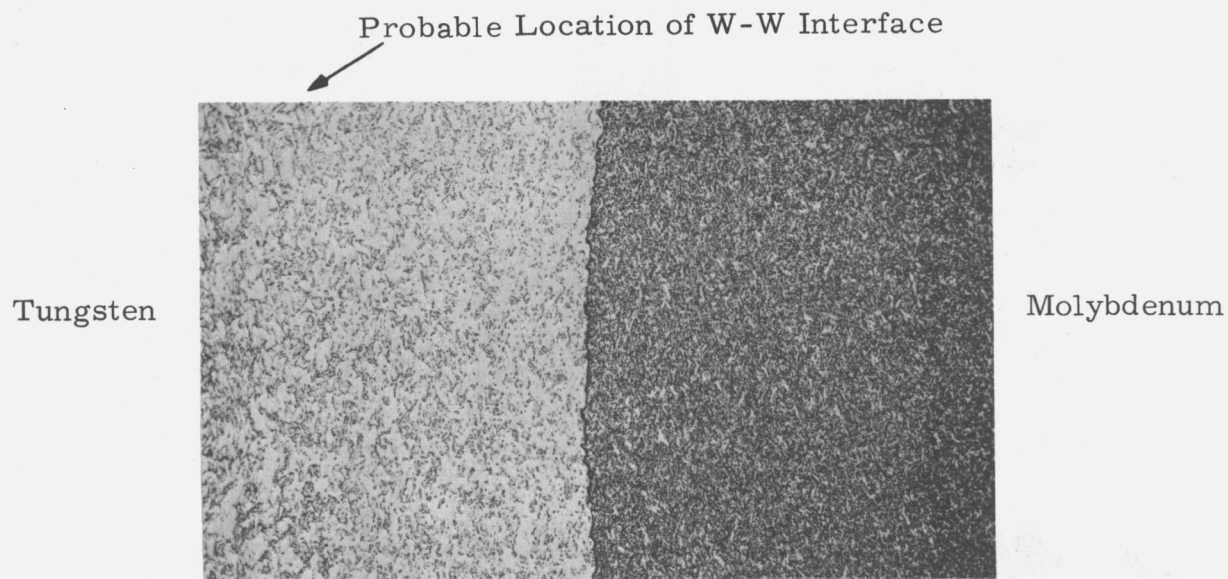
As-Extruded Surfaces of MoW-1, 2, and 3



Neg. 4Z2801B

FIGURE 8

Transverse Section of MoW-1 Near the Front
50 X (Murakami's Etch)



Neg. 4Z-280 2A

FIGURE 9

Transverse Section of MoW-1 Near the Rear
50 X (Murakami's Etch)

extrusion was 210 tons. The ZrO_2 coated die performance was excellent. The die orifice increased only 0.018 in. in diameter and was in satisfactory condition for additional extrusions (Figure 10). The increase in die diameter apparently occurs at the start of the extrusion because the extrusion varies only 0.007 in. with the largest diameter at the front. The surface of the extrusion showed no evidence of the melting reaction experienced with MoW-1, but it was extremely rough as shown in Figure 7(b). Apparently the pyrolytic graphite used as the lubricant and thermal protection is too strong to flow properly. It breaks up in large chunks which imbed into the surface of the extrusion to cause the roughness. Metallographic samples taken from the front and rear of the extrusion showed the tungsten to have a fine equiaxed grain structure at the front and an extremely fine elongated grain structure at the rear. Complete W-W bonding was revealed in all but the front 8 to 9 in. of the extrusion. At the front, cracks are evident in both the W-W interface and radially within the tungsten. The rest of the extrusion showed no evidence of cracks. The structures and tungsten bonding are essentially identical to those of MoW-3 which are shown in Figures 11, 12, and 13.

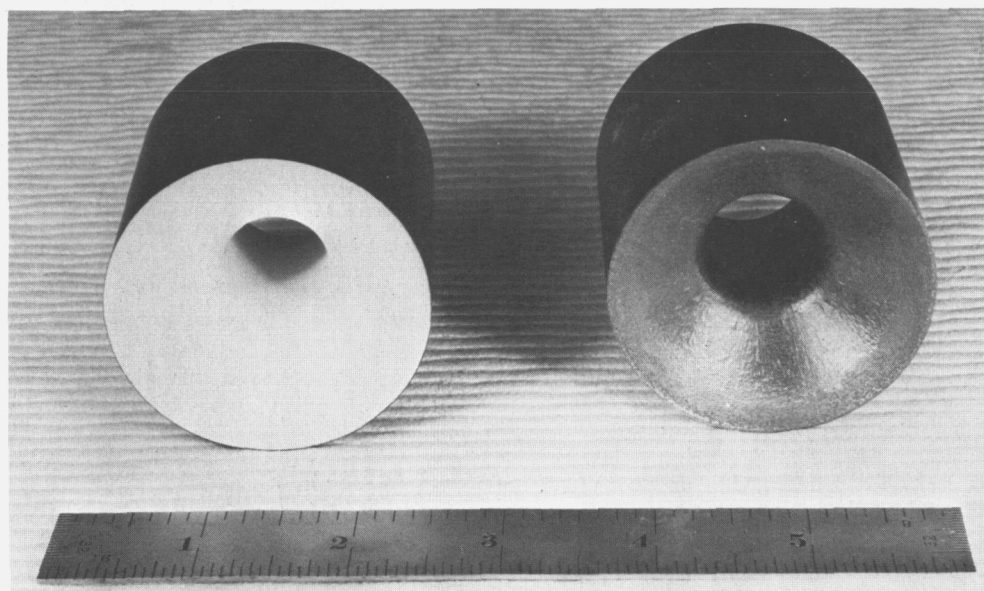


FIGURE 10

ZrO_2 Coated Die Before and After Extrusion of MoW-2

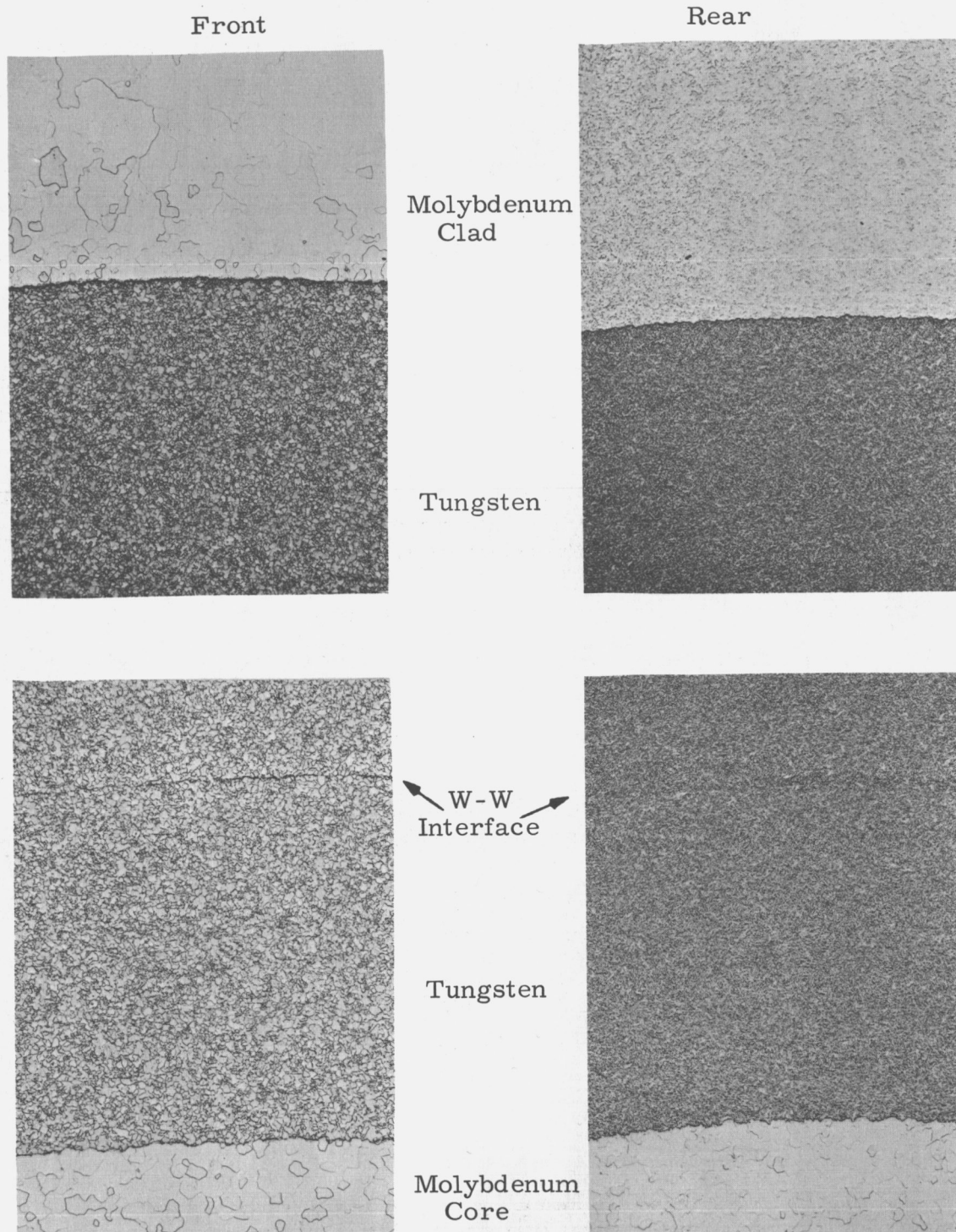


FIGURE 11

Transverse Sections of MoW-3
50 X (Murakami's Etch)

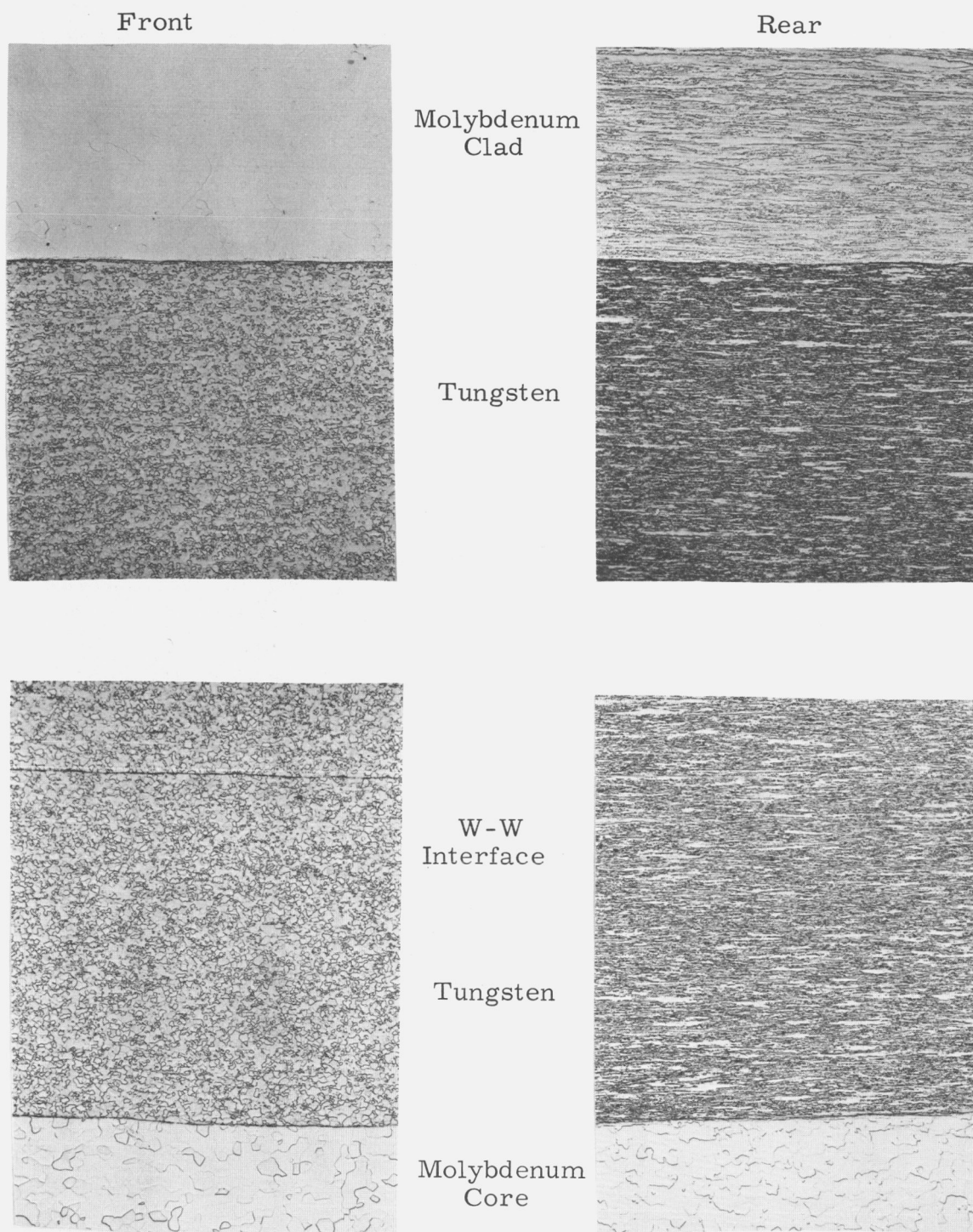
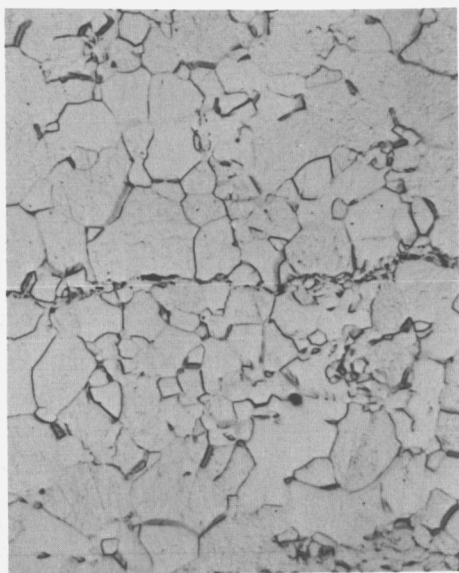
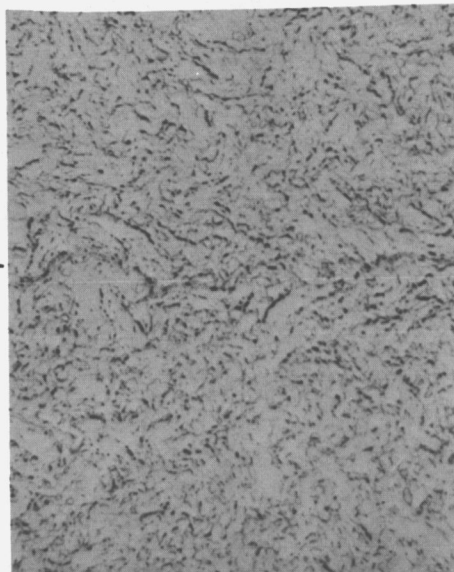


FIGURE 12
Longitudinal Sections of MoW-3
50 X (Murakami's Etch)

Front

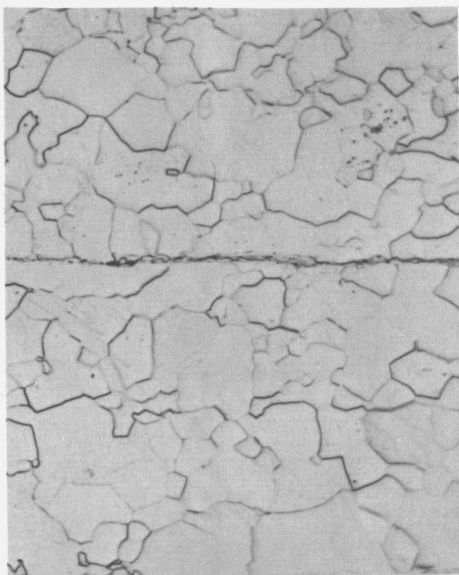


Back



Tungsten
Interface

Transverse



Tungsten
Interface



Longitudinal

FIGURE 13

W-W Interface of MoW-3
500 X (Murakami's Etch)

Previous experience in the extrusion of Zircaloy-2 had shown that the use of a plain graphite sleeve for lubricant resulted in a good extruded surface as shown on the extrusion butt in Figure 14. A Zircaloy-2 billet

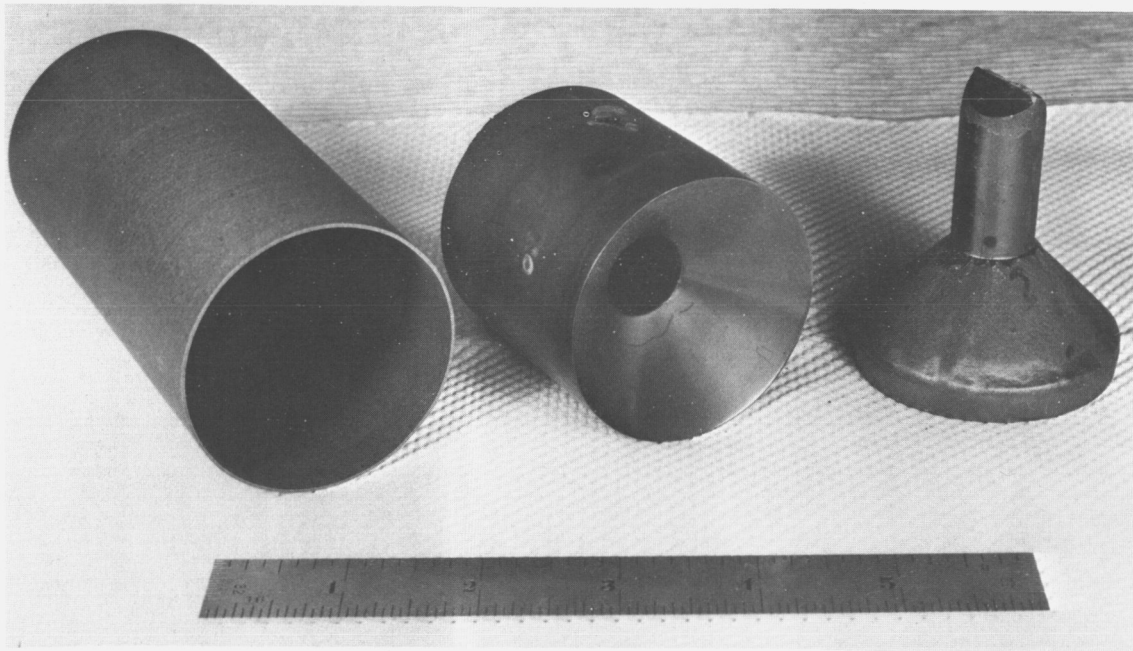


FIGURE 14

Graphite Sleeve and Die (after extrusion) Used for Zircaloy-2 Extrusion

(The butt is shown on the right. The excellent surface on the butt is typical of the full extrusion.)

was extruded in pyrolytic graphite to obtain a comparison with the use of plain graphite. The extruded surface (Figure 15) was nearly identical in degree and type of roughness with that of tungsten sleeve extrusion, MoW-2. Based on this information it was decided to try a plain graphite sleeve for the third tungsten sleeve extrusion. The billet components for MoW-3 were cleaned and assembled in the same manner as MoW-2, and it was extruded with an 1800 °C preheat and a plain graphite lubrication sleeve. The maximum force required during the extrusion was 210 tons. The surface of the extrusion (shown in Figure 7(c)) was much improved over extrusion MoW-2. The W-W bonding and microstructure of the

extrusion are similar to that of MoW-2 with little or no cracking at the front. Figures 11, 12, and 13 show the microstructure of MoW-3. The tungsten grains were equiaxed at the front and elongated, recrystallized, and much finer at the rear. The molybdenum cladding at the front showed a duplex grain size with a band of fine grains at the outer surface and large grains near the tungsten. This changed to a uniform, finer grain at the rear. Detailed examination of the structure shows a definite temperature gradient from front to rear of the extrusions. Bonding improvement is associated with the fine, elongated, recrystallized structure in the rear, cooler portion of the extrusion. The bond region was discernible only at high magnification and then only by following the "dirt" which was apparently present on the original surfaces.

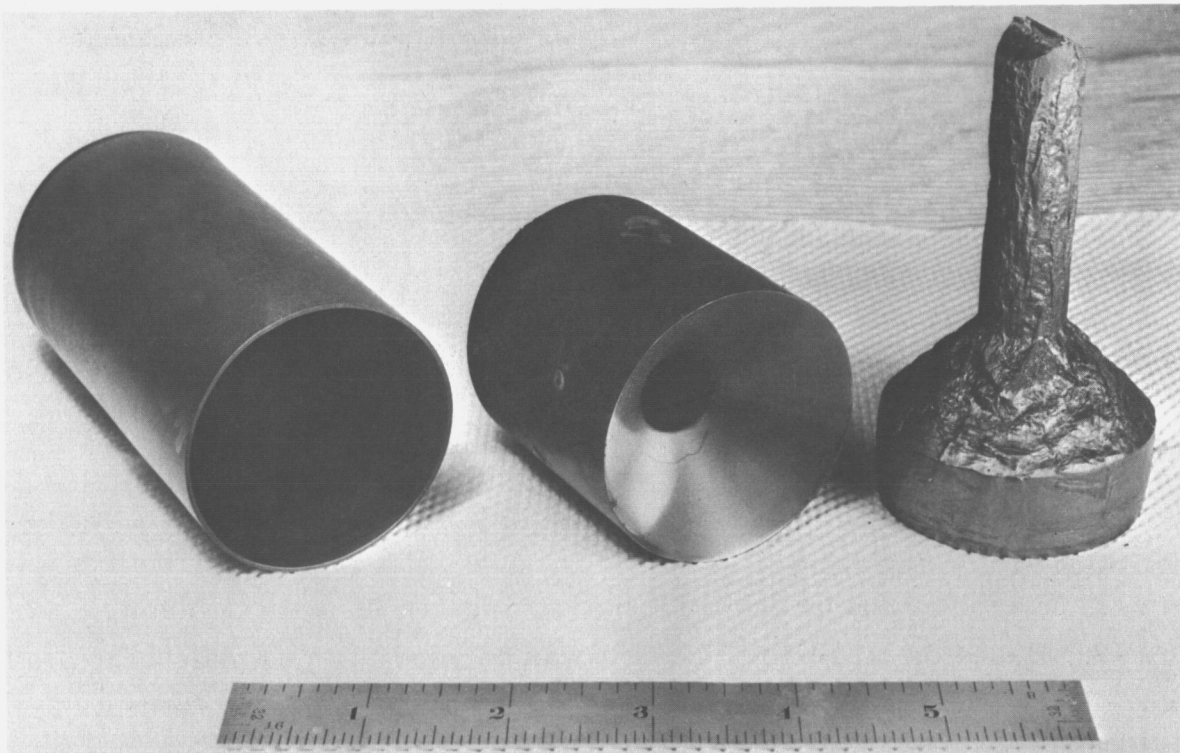


FIGURE 15

Pyrolytic Graphite Sleeve and Die Used for Zircaloy-2 Extrusion
(Surface on butt shown at right is typical for the full extrusion.)

The overall success of extrusion MoW-3 prompted changing to a more complicated geometry for extrusion number four. This was done to determine the ability of the process to reproduce a more complex billet cross section in the extrusion. Materials were immediately available to modify the shell-in-shell geometry to the tungsten plate-in-shell cross section shown in Figure 16. The slots were formed in the ID of the sintered

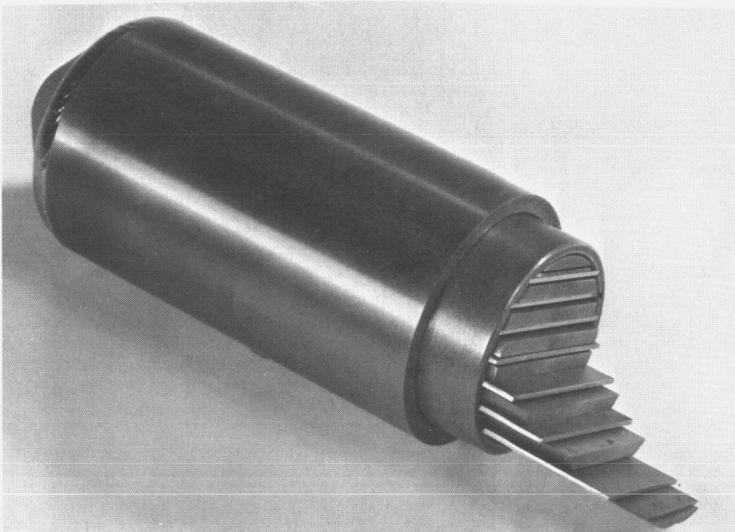


FIGURE 16
Plate-in-Shell Billet
Used for Extrusion
MoW-4

tungsten shell by spark discharge machining. Rolled and stress relief annealed tungsten plates 0.045 in. thick were machined and placed in the slots. The spaces between the plates were filled with 0.030 and 0.125 in. thick rolled and stress-relieved molybdenum plate. The tungsten shell and plates were degreased, assembled, and hydrogen fired 1 hr at 900 °C for cleaning. The molybdenum components were cleaned by dry grit blasting prior to assembly and vacuum electron beam closing of the billet. The extrusion, MoW-4, was performed with an 1850 °C preheat, ZrO₂ coated die, and no graphite sleeve. The omission of the graphite sleeve with only an oil-graphite dispersion in the container was done to observe the effect of extruding the bare molybdenum. The ZrO₂ coated die performed adequately and the entire extrusion surface was as smooth as the

the front half of the middle section of MoW-3, shown in Figure 7(c). A very slight washing or scoring of the container wall occurred. The extrusion force varied from 220 to 310 tons.

Metallography of a center cross section showed a good diffusion bond with some oxide inclusions between the tungsten shell and plates. No cracks were evident at any point in this section. Figure 17 shows the wrought tungsten and molybdenum components produced a larger grain size than the sintered components did in the extrusion. As in all previous extrusions the grain is finest toward the rear of the extrusion. The effect of the large grain in the wrought material was to wrinkle its interface with the adjacent material. The larger grain in the wrought versus as-sintered material is due to increased growth rate during the billet preheat. The molybdenum was removed from short section, about 1 in. long, by dissolution in hot $1/3 \text{ H}_2\text{O} - 1/3 \text{ H}_2\text{SO}_4 - 1/3 \text{ HNO}_3$ acid mixture. As seen in Figure 18, the plates, disregarding wrinkling, remained fairly straight and the shell remained round. Variation from the billet geometry is due, in large part, to the radial asymmetry of the plate design.

The fifth extrusion experiment, MoW-5, was designed to demonstrate the extrusion of a 3 in. diameter tungsten honeycomb composite billet. Arc vacuum cast and rolled, 5/16 in. diameter molybdenum rod was warm drawn through a hexagonal Turk's head to 0.243 in. across the flats. This rod was coated with 0.025 in. thick tungsten by the WF_6 vapor deposition process and cut to 4 in. lengths. A bundle of 37 of these rods was placed in a 3 in. OD wrought AVC molybdenum can which was machined to closely fit the bundle as shown in Figure 19. The billet was assembled with no cleaning of the as-deposited tungsten surface, degreasing only of the as-machined molybdenum, and closed by vacuum electron beam welding. It was extruded at 1800 C preheat with no lubricant, other than oil-graphite in the container, at 60 in./min ram speed. The ZrO_2 coated, 90° cone die had a 1.030 in. diameter orifice for a 9:1 extrusion ratio. Maximum force was 480 tons. As with MoW-4 the surface of the extrusion was excellent, but the container sustained a slight amount of scoring. Examination of

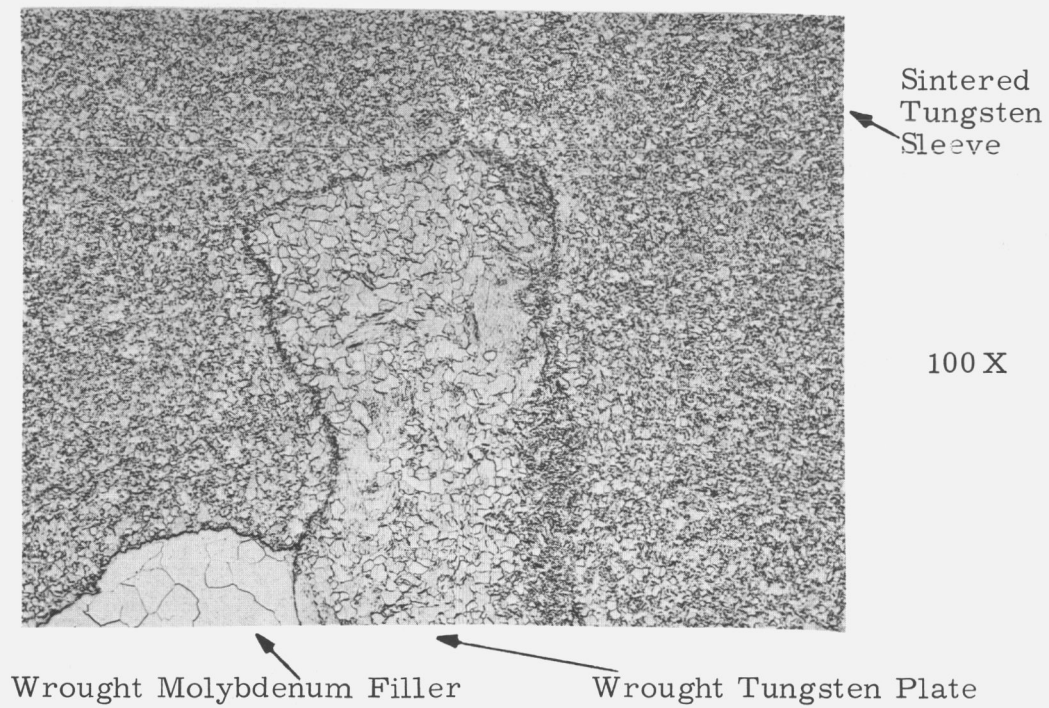
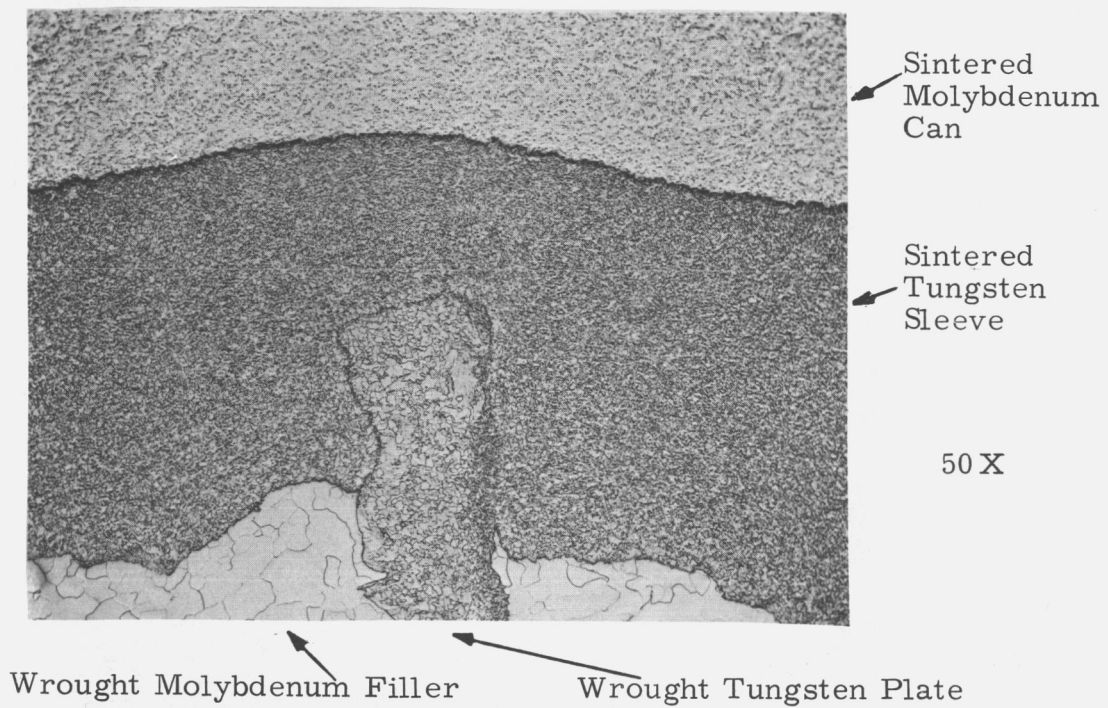
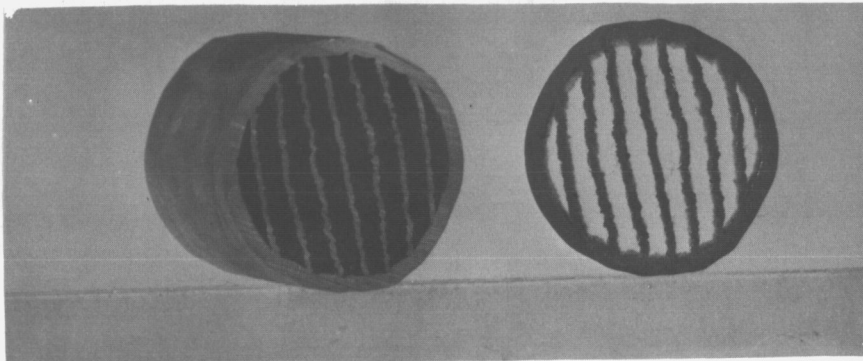


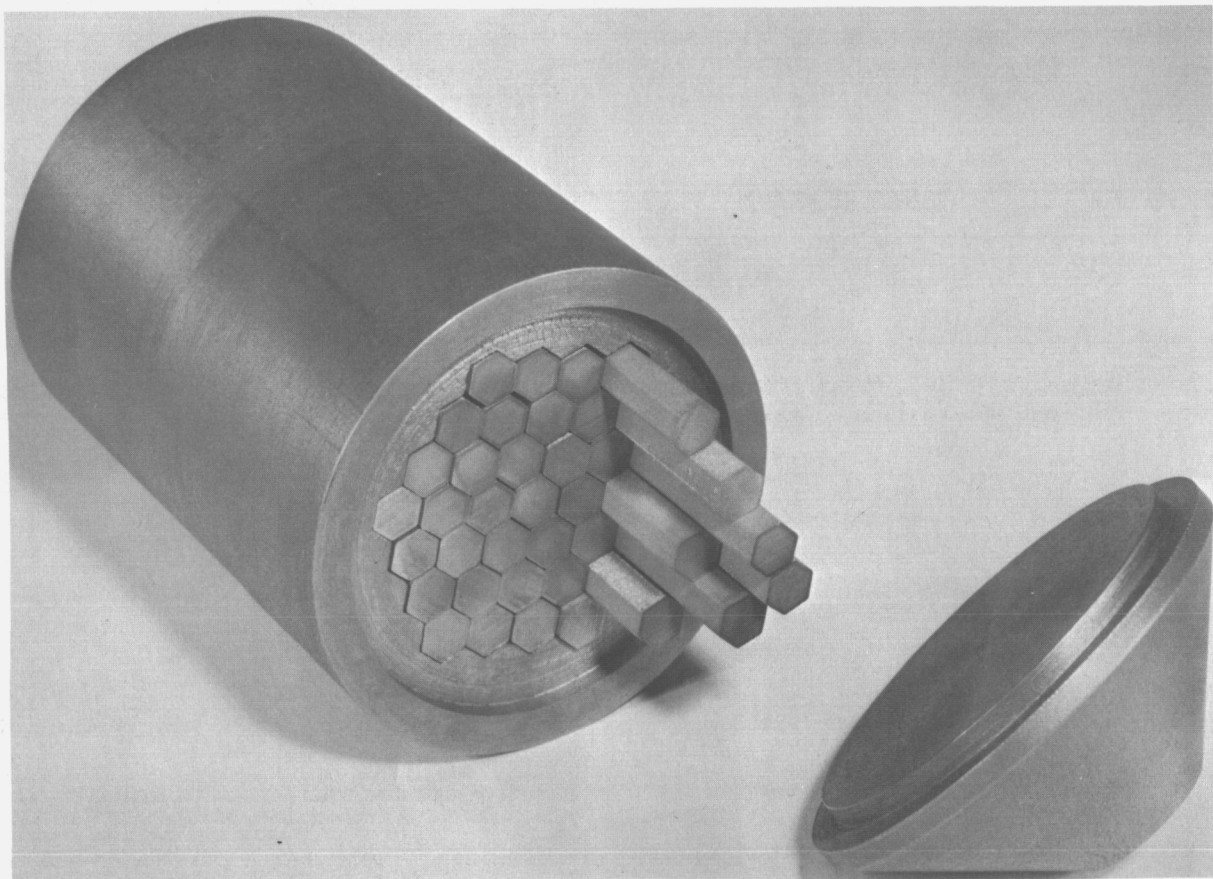
FIGURE 17
Central Transverse Section of MoW-4 (Murakami's Etch)



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FIGURE 18

MoW-4 After Removal of the Molybdenum



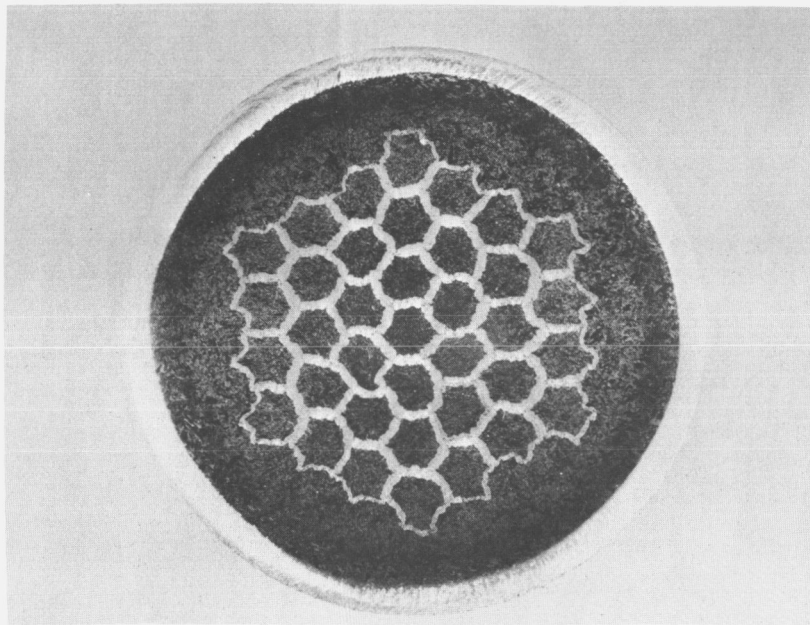
Neg. 0640116

FIGURE 19

~0.9X

Billet from Which MoW-5 was Extruded

the central cross section shown in Figure 20 revealed excellent W-W bonding, but wrinkled W-Mo interfaces and distortion of the cells from hexagonal shape. Both the wrinkling and cell distortion were due to large grained molybdenum and large columnar grains in the as-deposited tungsten. Figure 21 shows the transverse and longitudinal structures of the as-coated rod. A 2-7/8 in. length of the honeycomb (shown in Figure 22) from which the molybdenum was dissolved revealed no twisting of the extrusion. The web thickness is approximately 0.015 in. between cells and 0.0075 in. on the outside.

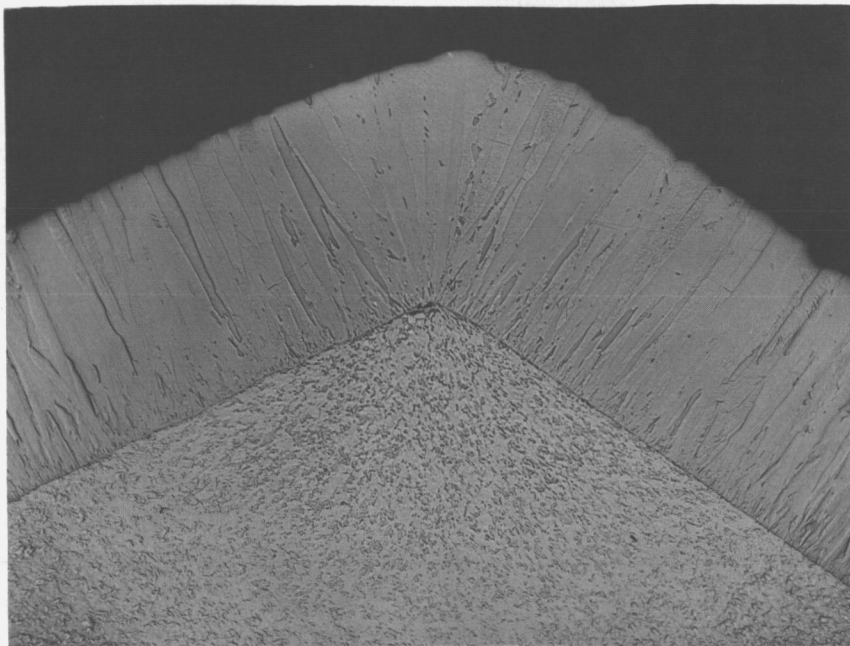


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FIGURE 20

Section of MoW-5 Near Center Which Shows Distortion of Web
Typical of the Full Extrusion

CONFIDENTIAL



Transverse

Tungsten Coat

Molybdenum
Rod

Neg. 4Z4345E



Longitudinal

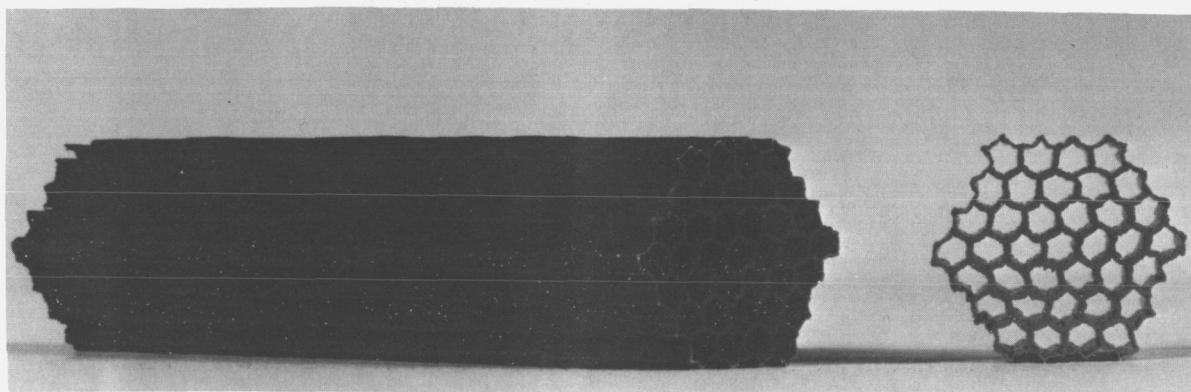
Tungsten Coat

Molybdenum
Rod

Neg. 4Z4345D

FIGURE 21

Hexagonal Rod Used in MoW-5 as WF_6 Vapor Coated
(Murakami's Etch) 50 X



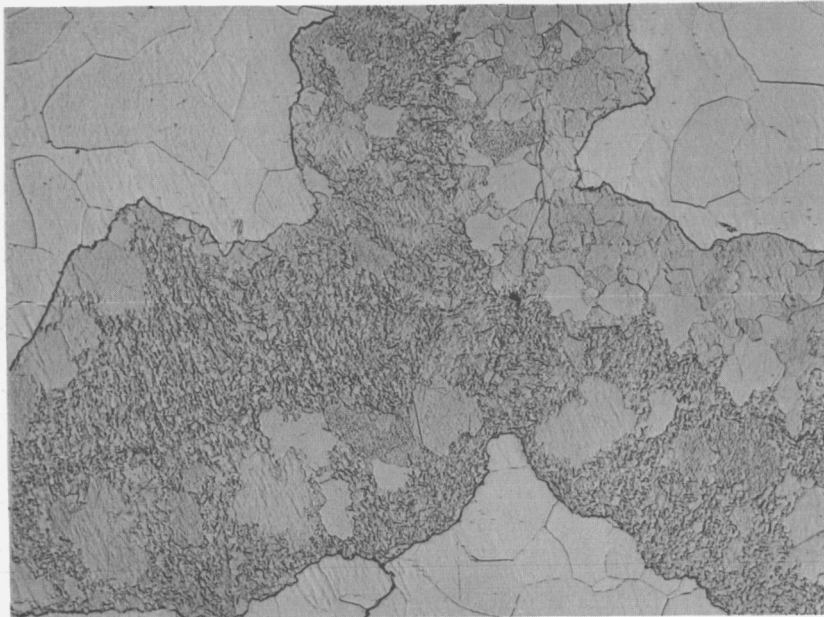
Neg. 0643118

FIGURE 22

A 2-7/8 in. Length of MoW-5 from Which the Molybdenum
Has Been Removed ~2X

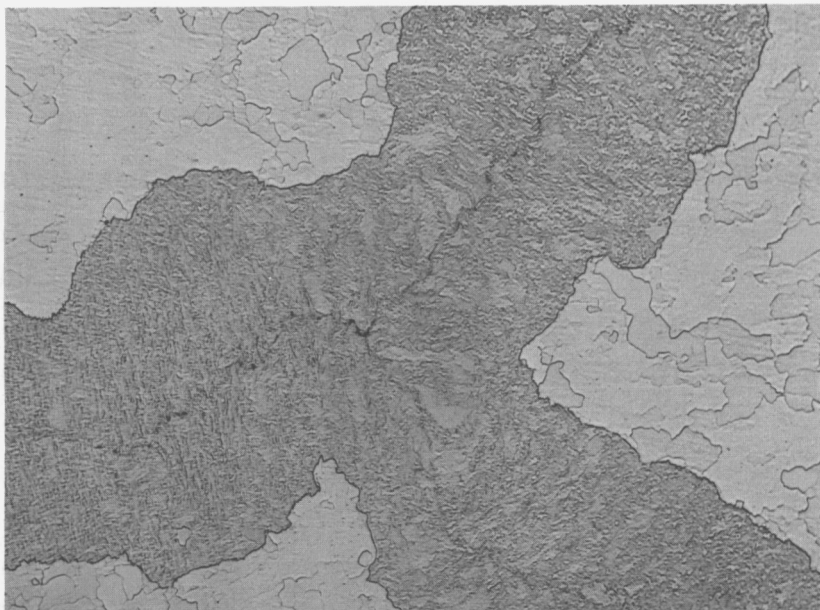
The structure of the tungsten and molybdenum is essentially the same as the wrought components of MoW-4. Figure 23 shows the structure at web intersection of front and rear transverse sections. The large grained tungsten in the upper right quadrant of the front section is not typical of this section and must be due to some difference in the original coating.

At this point sufficient data and experience had been gained to proceed with the design and procurement of components for the final unfueled tungsten honeycomb shown in Figure 3. The billet consisted of a 4 in. OD molybdenum can, the ID of which was coated with tungsten, six tungsten pieces to fill the spaces between the hex rod bundle and the round can, and a bundle of sixty-one hexagonal molybdenum rods coated by coextrusion with 0.030 in. thick tungsten. The coated rods were 0.400 in. across the flats and 6 in. long. Consultation with a vendor who was routinely fabricating similar material by coextrusion indicated no serious problems were involved in the production of the rod. An order was placed with them for 40 ft of the rod. Three feet of the material was received and appeared to be of excellent quality. Later information from



Front

Neg. 4Z3988A



Rear

Neg. 4Z3989A

FIGURE 23

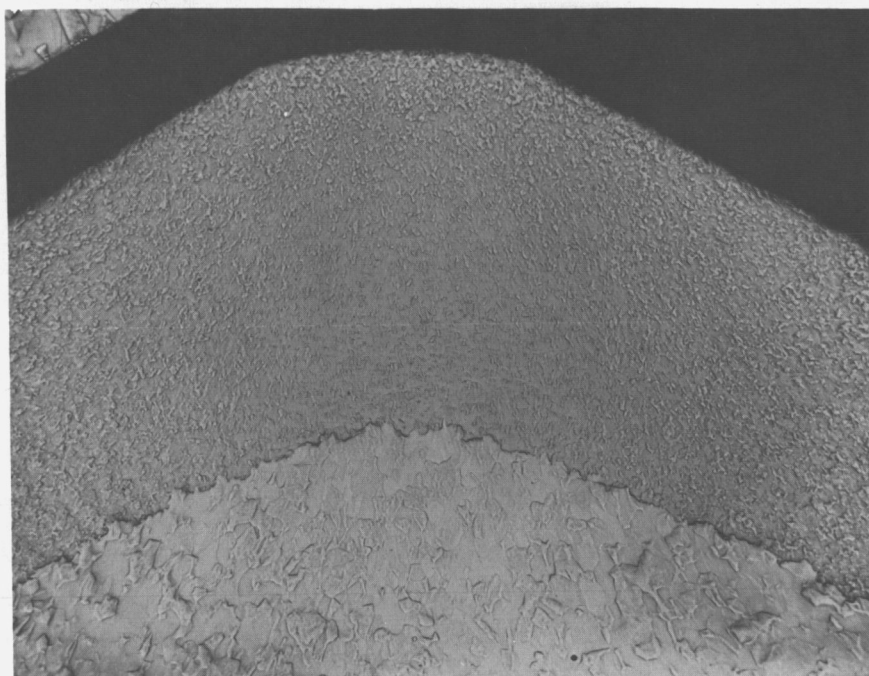
Transverse Sections at Web Intersections of MoW-5
(Murakami's Etch) 100 X

the vendor stated this was all the material to be furnished on the order due to technical difficulties in its production. Termination of this program precluded reordering the material. Figure 24 shows the transverse and longitudinal structure of the rod which was received. The relatively fine grain structure in both the tungsten and molybdenum and the smooth interface reflects the use of sintered components from which the rod was coextruded. The material used as honeycomb billet components (because of the improved microstructure) might reduce or eliminate the web wrinkling and cell distortion experienced in MoW-5.

A 3 in. diameter billet was designed to demonstrate the extrusion of a hexagonal array of 37 honeycomb cells with a circular outer clad. The molybdenum rods and can ID were prepared and delivered to a vendor for coating with tungsten by electro-deposition from a molten fluoride salt bath. The bath to be used became contaminated with nickel which deposited in the tungsten coating in a prohibitive concentration. The coating of the billet components was held up pending purification of the bath, and they were not received until after program termination. Both the coating surface and dimensions of the parts dictated that an expensive and time consuming grinding operation would be required to get an adequate billet assembly.

Experiments to obtain smooth W-Mo interfaces utilizing extrusion of as-compacted powder components were performed. These are described in Table I (p. 6) as Powders 1-3. Tungsten powder compacts of 70% theoretical density and 0.75 in. diameter were canned in 2.25 in. diameter mild steel and 304 stainless steel cans. These billets were extruded at 9:1 ratio and preheats varying from 1100 to 1200 °C. In all, the tungsten compact broke into many pieces distributed in the length of the extrusion, and sustained little or no reduction in area. The gross mismatch of can and core stiffness plus the thick, soft can wall accounts for this.

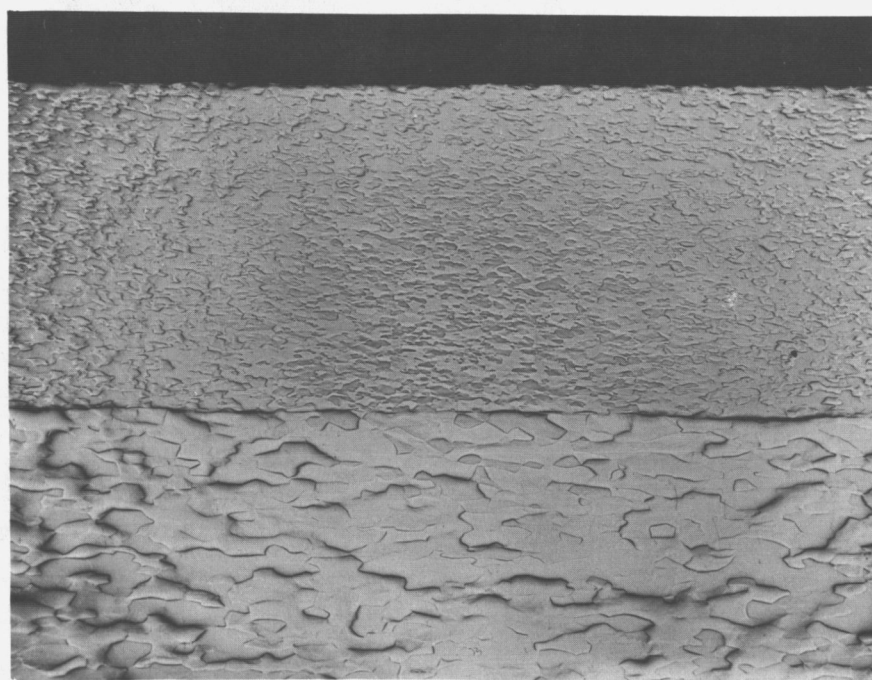
A billet (Powder 4 in Table I, p. 6) of the same geometry with an as-sintered molybdenum can and a 75% TD as-compacted, coprecipitated, submicron W-10 wt% UO_2 core³ was extruded at 9:1 ratio and 1875 C

Transverse

Tungsten

Molybdenum

Neg. 4Z4344A

Longitudinal

Tungsten

Molybdenum

Neg. 4Z4344F

FIGURE 24

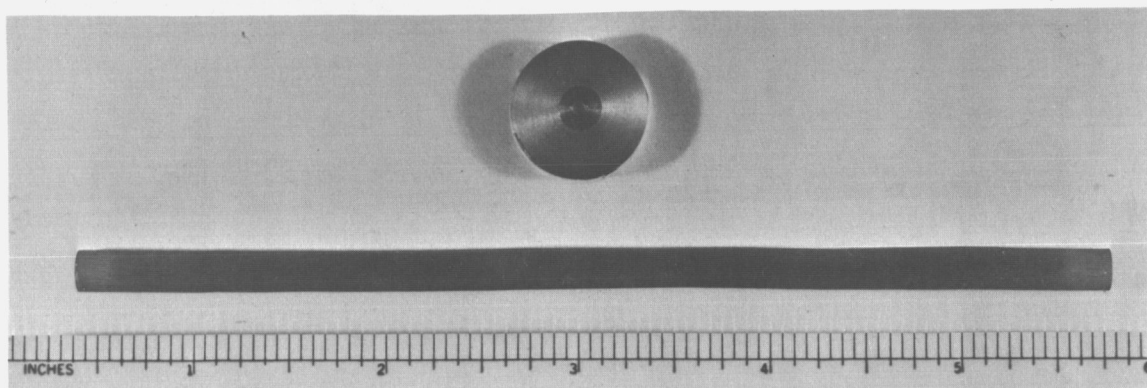
Hexagonal Molybdenum Rod Coextrusion Clad with Tungsten,
0.400 in. Across Flats
(Murakami's Etch) 50 X

UNCLASSIFIED

preheat. Figures 25 and 26 show the microstructure of a central section of the extrusion. As seen in the 1X macrophoto in Figure 25, the interface between the W- UO_2 core and as-sintered molybdenum can is smooth. The core exhibited no porosity indicating near theoretical density was achieved. Nearly all the UO_2 particles are less than 5μ transverse dimension and 45μ in length with very little interconnection. The tungsten grains are somewhat shorter than the UO_2 particles and exhibit an elongated partially recrystallized shape. The estimated UO_2 particle size was 2 to 4μ prior to billet preheat and 6 to 7μ after preheat. The latter is calculated back from the as-extruded size on the basis of the extrusion ratio.

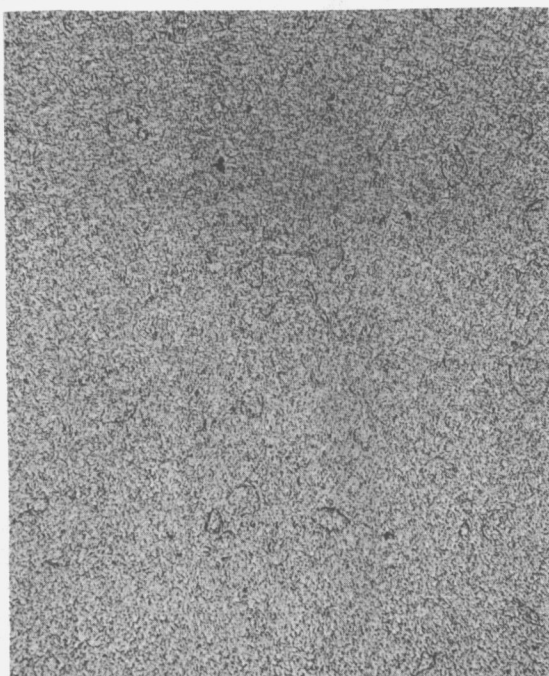
Another experiment (Powder 5 in Table I, p. 6) utilized cocompaction of powders to form the billet core. A 7-rod array of hexagonal steel rods were suspended in a hollow cylindrical rubber mold, and the spaces between the rods and between the array and mold were packed with tungsten powder to 30% TD. As the steel rods were withdrawn, one at a time, the resulting cavities in the tungsten powder were packed with molybdenum powder. Rubber end plugs were placed in the mold which was then pressed cold in an extrusion container at 80,000 psi to about 70% TD. This was canned in molybdenum and extruded at 9:1 ratio and 1800°C preheat. The billet stalled after about 8 in. of good extrusion. The cross section of the extrusion exhibited smooth W-Mo interfaces with irregularities in cell shape due to crude jiggling in the cocompacting step. Figure 27 shows cross sections as extruded and with the molybdenum removed. Density measurements indicated the tungsten in both the extrusion and butt after removing the molybdenum was 100% of theoretical.

A possible practical solution to the slight container damage when extruding bare molybdenum is indicated by an experiment with a ceramic coated liner. A 3.050 in. diameter liner of H-21 tool steel was flame sprayed with ZrO_2 , 0.035 in. thick. It was placed in a 3.550 in. diameter container with a light shrink fit. Two, three inch diameter, 304 stainless steel billets (Coated Cont. 1 and 2 in Table I, p. 6) were extruded from this liner at 9:1 ratio and preheats of 1150 and 1250°C . Graphite sleeves



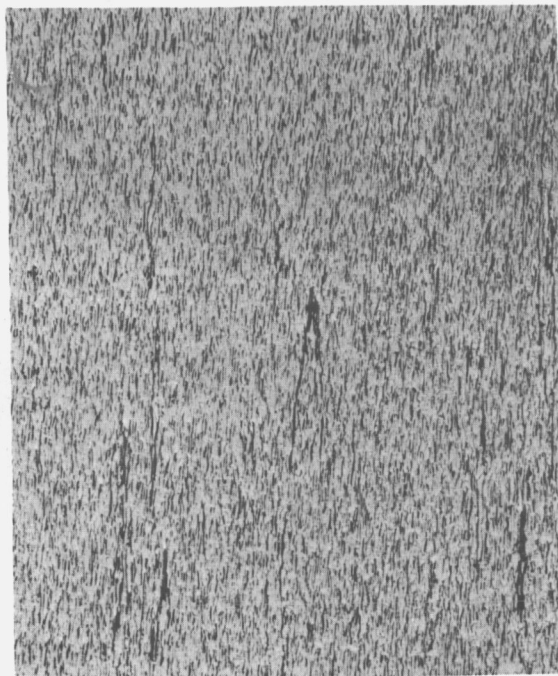
Neg. 5Z-5094

1X

Transverse Section
Neg. 4Z-4592A

100 X

As-Polished



Longitudinal Section

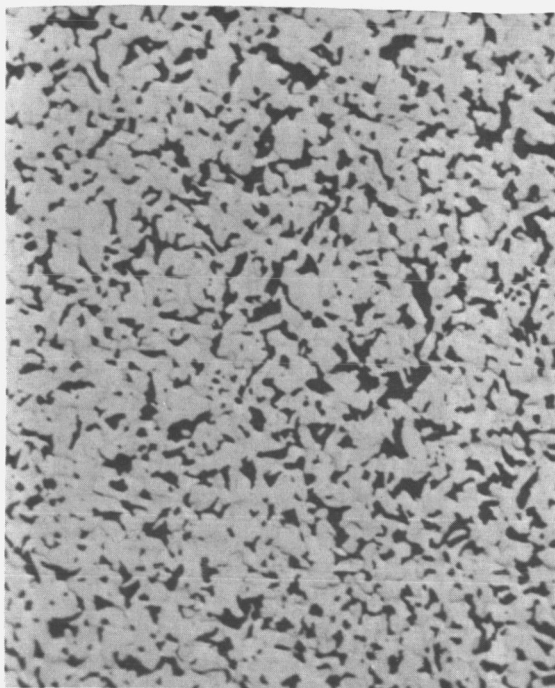
100 X

Neg. 4Z-4594A

FIGURE 25

W-10 wt% UO_2 Extruded from Green Compact
Upper: As-extruded section showing molybdenum can
Lower: Molybdenum chemically removed

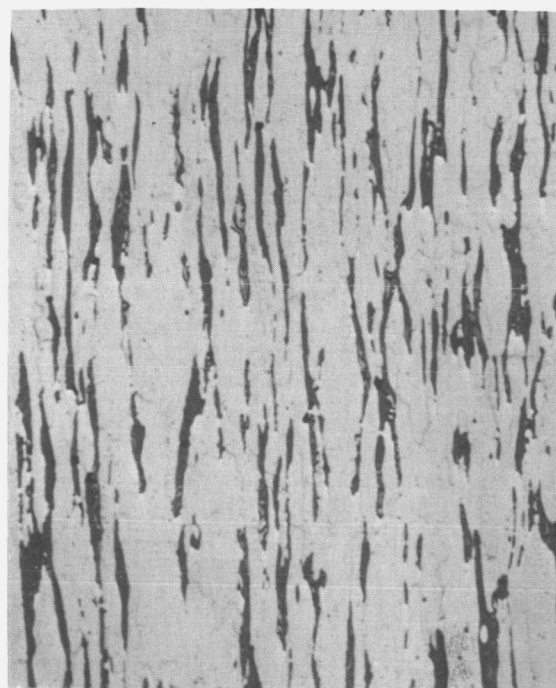
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Transverse Section
Neg. 4Z-4592B

1000 X

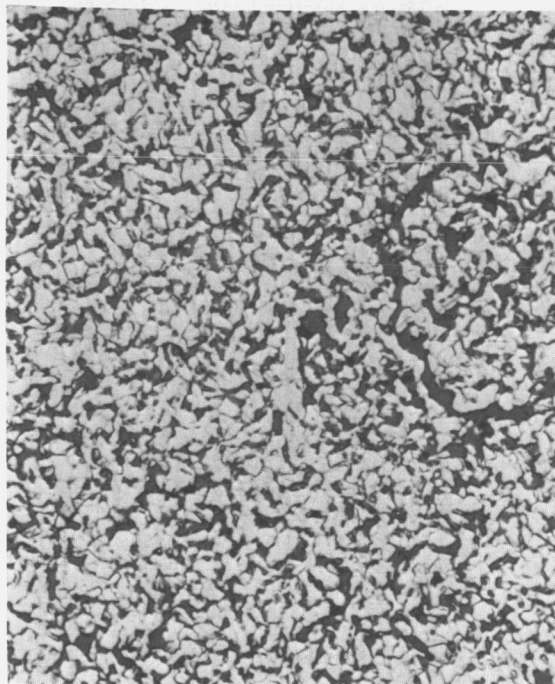
As-Polished



Longitudinal Section

1000 X

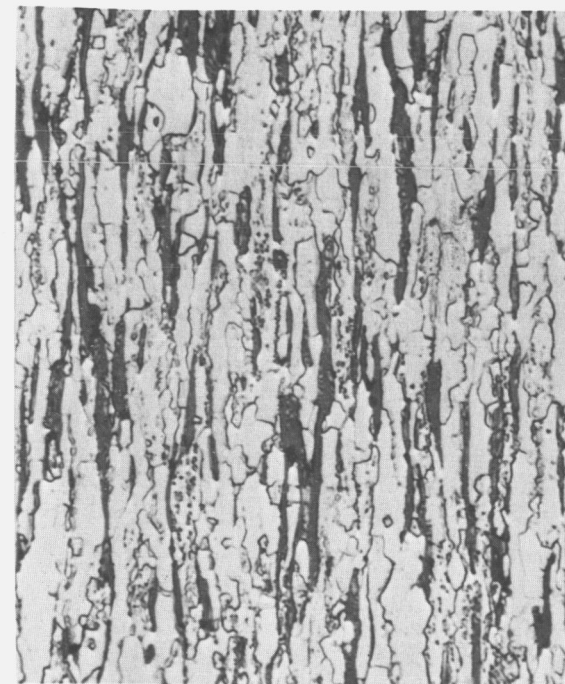
Neg. 4Z-4594B



Transverse Section
Neg. 4Z-4592D

1000 X

Etched (Murakami's etch)



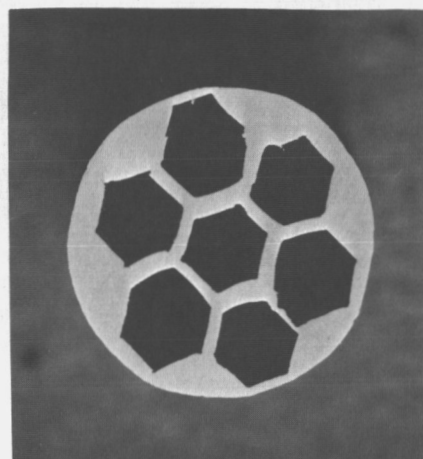
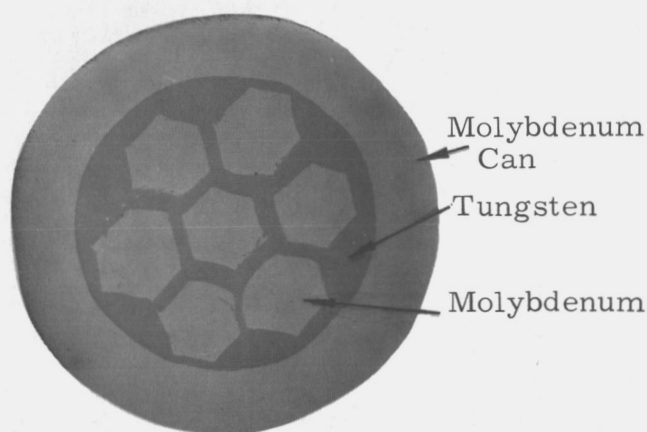
Longitudinal Section

1000 X

Neg. 4Z-4594B

FIGURE 26

As-Extruded W-10 wt% UO_2 Compact

As-Extruded

Molybdenum Removed

FIGURE 27

Cocompacted Tungsten and Molybdenum Powders
Extruded in a Sintered Molybdenum Can 3X

were used for billet lubricant. Maximum container pressure was 59 tons/in.². The surfaces of the extrusions were excellent, even where the graphite had been displaced and bare steel was in contact with the ZrO_2 on the liner and die. No damage to the container liner was observed and it was usable for additional extrusions. The liner was easily removed after the extrusions were completed.

DISCUSSION

The wrinkling of the tungsten web and associated cell distortion which occurred in the plate-in-shell (MoW-4) and honeycomb (MoW-5) extrusions is the obvious major problem in fabricating tungsten honeycomb by coextrusion. Since it is due to asymmetric flow of large grains in one or more of the billet materials, the solution lies in maintaining a fine grained structure in the billet through preheat. Billet components of as-sintered tungsten and molybdenum meet this requirement judging from the smooth interfaces observed in extrusions in which they were used. Fabrication of tungsten clad, hexagonal molybdenum rods in the as-sintered condition within the

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close dimensional tolerances required is feasible, but difficult and costly. The ultimate goal of honeycomb with UO_2 -fueled web which is clad with pure tungsten would require molybdenum rod components clad with a thin layer of tungsten in addition to the fuel layer. This is considered impractical, if not impossible, to fabricate by compacting and sintering. However, green powder extrusion (extrusion of green lubricated powder) offers a possibility of fabricating the billet rods in the as-sintered condition. The green composite extrusion billet could be made of a cylinder of molybdenum surrounded by shells of tungsten and the W- UO_2 fuel and coextruded to green hexagonal rod. Sintering followed by a sizing and straightening step should furnish rods suitable for honeycomb coextrusion billet components.

The original plan utilizing coextrusion to produce the hexagonal rods for the honeycomb billet core has a good possibility for successful production of a clad, fueled-web honeycomb. The work done by the vendor attempting the coextrusion of the tungsten clad hexagonal rod established a good feasibility for the process. The small quantity of rod completed was of excellent quality and structures which are believed to be adequate for subsequent coextrusion of good quality honeycomb. A fueled section would require W- UO_2 clad with tungsten. Use of these materials in the original coextrusion should present no major problem. The tungsten clad could be applied to the molybdenum core of the rod billet by vapor deposition.

The use of submicron W- UO_2 in one 9:1 ratio extrusion produced a fully dense structure in which the UO_2 stringer length was below 45μ . The nature of the UO_2 stringering in this structure after the second of the two extrusions required in the honeycomb process can only be postulated at this time. Since there was little agglomeration of the UO_2 in the one extrusion attempted, it is entirely possible that re-extrusion would not result in the interconnected type stringering which is considered undesirable. Possible excessive grain growth in the honeycomb billet components would be controlled by the addition of minor alloying constituents to the billet materials.

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The longitudinal variation in the grain size of both the tungsten and molybdenum observed in all the extrusions is due entirely to the billets chilling in the container. The major cause of this is the low ram speed (60 in. /min) of the press used. Presses are available with at least ten times this speed which should practically eliminate the longitudinal structure variation. The use of ceramic coated container liners would be a considerable aid in reducing the chilling of the billet. Both increased ram speed and coated container liners would permit a lower billet preheat temperature and tend to minimize the growth of large grains in the billet components.

CONCLUSIONS AND RECOMMENDATIONS

The following conclusions can be drawn from the work described in this report:

1. Excellent W-W solid-state diffusion bonds can be produced by coextrusion at 1800 °C and 9:1 extrusion ratio.
2. Tungsten honeycomb structure with continuous defect-free web at least as thin as 0.008 in. can be fabricated by coextrusion with molybdenum as sacrificial material.
3. Coextrusion of tungsten honeycomb with uniform cell shape and wrinkle-free web depends upon maintaining a fine grain size in all billet components through billet preheat.
4. Extrusion of 70% theoretical density tungsten powder compacts produces a full density wrought structure.
5. Extrusion of W-UO₂ submicron particle size 75% dense powder compacted at 1875 °C and 9:1 ratio results in a full density product with maximum UO₂ stringer length of 45μ.
6. Tungsten clad W-UO₂ honeycomb shape fuel elements can be coextruded with very large length to diameter ratios. The practical limit for the length of the finished element will be the time consumed in removing the sacrificial material.

It is recommended that future work be undertaken to:

1. Characterize the honeycomb fuel element resulting from the coextrusion of billets assembled from rod components fabricated by the coextrusion of billets containing submicron $W-UO_2$ and as-sintered molybdenum components.
2. Investigate the feasibility of forming tungsten-clad, $W-UO_2$ honeycomb billet rod components by green powder coextrusion of the materials followed by sintering and sizing. If successful, this method permits the use of all as-sintered fine grained components in the honeycomb billet.
3. Investigate the mechanical and fuel retention properties of extruded, submicron $W-UO_2$ powder compacts.

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